

AD A 044986

NSWC/WOL TR 76-81

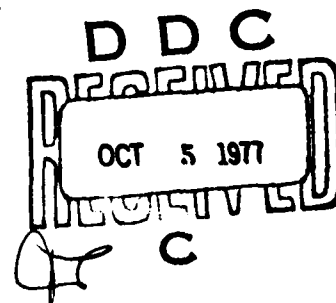
12  
B.S.

# NOMINAL 60-NITINOL EOD TOOLS - PRODUCTION INVESTIGATION AND EVALUATION

BY R. H. LUNDSTEN  
W. J. BUEHLER  
R. E. JONES

RESEARCH AND TECHNOLOGY DEPARTMENT

JUNE 1977



AD No. \_\_\_\_\_  
DDC FILE COPY



**NAVAL SURFACE WEAPONS CENTER**

Dahlgren, Virginia 22448 • Silver Spring, Maryland 20910

DISTRIBUTION STATEMENT A  
Approved for public release;  
Distribution Unlimited

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

| REPORT DOCUMENTATION PAGE   |   | READ INSTRUCTIONS<br>BEFORE COMPLETING FORM |
|---|---|---|
| 1. REPORT NUMBER<br>14 NSWC/WOL/TR-76-81  | 2. GOVT ACCESSION NO.   | 3. RECIPIENT'S CATALOG NUMBER               |
| 4. TITLE (and Subtitle)<br>6 Nominal 60-NITINOL EOD Tools • Production Investigation and Evaluation   | 5. TYPE OF REPORT & PERIOD COVERED  |   |
| 7. AUTHOR(s)<br>10 R. H. /Lundsten, W. J. /Buehler, R. E. /Jones  | 6. PERFORMING ORG. REPORT NUMBER  |   |
| 9. PERFORMING ORGANIZATION NAME AND ADDRESS<br>Naval Surface Weapons Center<br>White Oak Laboratory<br>White Oak, Silver Spring, Maryland 20910   | 10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS<br>0; 0; SEA 333-501-004-1/<br>SF 34-373-302; 0 |   |
| 11. CONTROLLING OFFICE NAME AND ADDRESS   | 12. REPORT DATE<br>11 June 77   | 13. NUMBER OF PAGES<br>44                   |
| 14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)<br>12 48p.  | 15. SECURITY CLASS. (of this report)<br>UNCLASSIFIED  |   |
| 15a. DECLASSIFICATION/DOWNGRADING SCHEDULE  |   |   |
| 16. DISTRIBUTION STATEMENT (of this Report)<br><br>Approved for Public Release; Distribution Unlimited  |   |   |
| 17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)<br>16 F34373 17 SF34373302   |   |   |
| 18. SUPPLEMENTARY NOTES   |   |   |
| 19. KEY WORDS (Continue on reverse side if necessary and identify by block number)<br>NITINOL Tools<br>EOD Tools<br>EOD Swimmer Equipment<br>NITINOL Shell Moldings   |   |   |
| 20. ABSTRACT (Continue on reverse side if necessary and identify by block number)<br>An investigation of several methods for producing nominally 60-NITINOL Explosive Ordnance Disposal (EOD) tools is described. Three tool types were produced by shell mold casting techniques and are mechanically evaluated by comparison with similar tools of forged steel and beryllium-copper. |   |   |

DDC  
OCT 5 1977  
C

DD FORM 1 JAN 73 1473

EDITION OF 1 NOV 65 IS OBSOLETE  
S/N 0102-014-6001

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

391596

16

27 June 1977

NSWC/WOL/TR 76-81

NOMINAL 60-NITINOL EOD TOOLS - PRODUCTION INVESTIGATION AND  
EVALUATION

This report describes an investigation of methods for producing nominally 60-Nitinol Explosive Ordnance Disposal, (EOD) tools. Three tool types were produced by shell mold casting and evaluated by comparison with similar tools of forged steel and beryllium-copper. This report documents work performed by the Naval Surface Weapons Center, White Oak Laboratory as a portion of the Naval Sea Systems supported program for research in Low Magnetic Materials and Magnetic Effects for EOD (Task Number SEA333-501-004-1/SF34-373-302). This document is for information only.

*Paul R. Wessel*

PAUL R. WESSEL  
By direction

|                                  |   |        |
|----------------------------------|---|--------|
| ACCESSION                        |   |        |
| NTIS                             |   |        |
| DDC                              |   |        |
| UNANNOUNCED                      |   |        |
| JUSTIFICATION                    |   |        |
| BY                               |   |        |
| DISTRIBUTION/AVAILABILITY STATES |   |        |
| Dist.                            | A | Avail. |
| A                                |   |        |

CONTENTS

|   | Page |
|---|------|
| I. INTRODUCTION . . . . .                               | 6    |
| II. METALLURGICAL AND CASTING CONSIDERATIONS . . . . .  | 9    |
| III. PATTERNS AND SHELL MOLDS . . . . .                 | 11   |
| IV. VACUUM CASTING . . . . .                            | 15   |
| V. ALLOY MICROSTRUCTURE . . . . .                       | 20   |
| VI. HEAT TREATMENT . . . . .                            | 28   |
| VII. POST-CASTING TOOL FINISHING AND ASSEMBLY . . . . . | 30   |
| VIII. TEST AND EVALUATION . . . . .                     | 34   |
| IX. CONCLUSIONS . . . . .                               | 43   |
| REFERENCES . . . . .                                    | 44   |

## ILLUSTRATIONS

| Figure |  | Page |
|--------|--|------|
| 1      | One half of the wrench pattern used to produce the wrench shell mold.  | 12   |
| 2      | A typical mold half used to cast the 60-NITINOL wrench.  | 13   |
| 3      | Shell-mold cast assembly of the 60-NITINOL wrench.   | 16   |
| 4      | Cast 60-NITINOL wrench   | 17   |
| 5      | Cast 60-NITINOL hammer   | 17   |
| 6      | Cast 60-NITINOL pliers   | 18   |
| 7      | Comparison of 60-NITINOL, beryllium-copper and steel pliers.   | 18   |
| 8      | Comparison photo showing beryllium-copper, steel and 60-NITINOL hammers.   | 19   |
| 9      | Photomicrographs taken in different, but "typical" sections of the shell mold "as cast" 60-NITINOL material.   | 21   |
| 10     | Specimens taken from the 60-NITINOL pliers melt and given subsequent heat treatments (argon atmosphere) at 980°C for varying time periods followed by furnace cooling. | 22   |
| 11     | Continued heating for a total of 144 hours at 980°C (argon atmosphere) of the 60-NITINOL pliers melt followed by a furnace cool.                                       | 23   |
| 12     | "As Cast" 60-NITINOL specimens taken from the cast discs.  | 24   |
| 13     | Disc specimens after a 48 hour heat treatment (argon atmosphere) followed by a furnace cool.   | 25   |
| 14     | Disc specimens after a 96 hour heat treatment (argon atmosphere) followed by a furnace cool.   | 26   |

ILLUSTRATIONS

| Figure |  | Page |
|--------|--|------|
| 15     | The effects of heat treating various shell mold cast 60-NITINOL specimens for increased time periods at 980°C in argon atmosphere. | 29   |
| 16     | Standard test fixtures with 60-NITINOL tools under load.   | 35   |
| 17     | Tested wrenches.   | 36   |
| 18     | 60-NITINOL cast pliers failure.  | 40   |

TABLES

| Table |  | Page |
|-------|--|------|
| I     | Comparison of "Wrought" and "Arc-Cast"<br>60-NITINOL Tensile and Hardness Properties | 7    |
| II    | Data on Fully Annealed 60-NITINOL and<br>Standard Hardened Steel Tools               | 31   |
| III   | 60-NITINOL Tool Flame Hardness Results   | 32   |
| IV    | Combination Wrench Mechanical Test Results   | 37   |
| V     | Pliers Mechanical Test Results   | 39   |
| VI    | Hammer Mechanical Test Results   | 42   |

## I. INTRODUCTION

For some time the explosive ordnance disposal (EOD) community has searched for materials that would perform mechanically like hardened and tempered steel and also possess the quality of being stably non-magnetic. This quest has led to the investigation of numerous non-ferrous metallic alloys. Some of the more promising of these materials were based upon copper (e.g. beryllium-copper, variations of R. Dean's Cu-Ni-Mn), cobalt (Haynes Alloy 25), titanium and aluminum. Although all were basically non-magnetic none possessed all of the desirable mechanical properties of the standard iron-base alloy tools.

Concurrent with the search for EOD tool materials a new non-magnetic alloy system was discovered and developed at the Naval Surface Weapons Center, White Oak Lab (formerly Naval Ordnance Laboratory). This system based upon the component metals nickel and titanium was given the generic name NITINOL. While various compositions possessed differing mechanical properties, the compositions with 58 to 62 weight per cent nickel were found to be capable of precipitation hardening to tool steel hardness levels without altering their non-magnetic character. Thus the generically-named alloy 60-NITINOL was born and factored into the long list of candidate EOD materials.

A summary of "wrought" and "arc-cast" mechanical properties is given in Table I of this report. An examination of Table I reveals the outstanding hardness increase capability, ranging from 29 to 60 Rockwell-C. These same data also point up the limited elongation of about 1% in the quench hardened condition.

In view of the possibilities of the nominal 60-NITINOL alloy system a program was undertaken to attempt to produce three selected EOD tools for preliminary evaluation. Initial efforts were directed to the close-die forging of tools possessing complex shapes, e.g. the adjustable end-wrench. This work was performed at the Diamond Tool and Horseshoe Company, Duluth, Minnesota and met with marginal



TABLE I  
COMPARISON OF "WROUGHT" AND "ARC-CAST" 60-NITINOL TENSILE AND HARDNESS PROPERTIES

| Alloy Preparation     | Mechanical Property       |                       |                |  | Average Hardness (R <sub>C</sub> ) |
|-----------------------|---------------------------|-----------------------|----------------|--|------------------------------------|
|                       | Ultimate Tensile Strength | Yield Strength        | Elongation (%) | Young's Modulus                                      |                                    |
| <u>Wrought Alloy</u>  |                           |                       |                |  |                                    |
| Hot extruded          | 1227MPa (178Ksi)          | *                     | < 1            | 105.4x10 <sup>3</sup> MPa (15.3x10 <sup>6</sup> psi) | 51                                 |
| Quenched              | 1062MPa (154Ksi)          | *                     | < 1            | 113.8x10 <sup>3</sup> MPa (16.5x10 <sup>6</sup> psi) | 60                                 |
| Furnace Cooled        | 945MPa (137Ksi)           | 352MPa (51Ksi)        | 7              | 97.9x10 <sup>3</sup> MPa (14.2x10 <sup>6</sup> psi)  | 35                                 |
| <u>Arc-Cast Alloy</u> |                           |                       |                |  |                                    |
| As Cast               | 552-965MPa (80-140+Ksi)   | *                     | < 1            | 96.5x10 <sup>3</sup> MPa (14x10 <sup>6</sup> psi)    | 54                                 |
| Quenched              | 827-965MPa (120-140Ksi)   | *                     | < 1            | 99.9x10 <sup>3</sup> MPa (14.5x10 <sup>6</sup> psi)  | 60                                 |
| Furnace Cooled        | 552-655MPa (80-95Ksi)     | 159-172MPa (23-25Ksi) | < 1            | 71.0x10 <sup>3</sup> MPa (10.3x10 <sup>6</sup> psi)  | 29                                 |

\*Yield strength not precisely measured, but approaches the value for ultimate tensile strength.

+Strength and other properties vary with cooling rate of casting.

success. 60-NITINOL bars were successfully forged into tools in the 825° to 850°C forging range but repeated very high energy blows were needed to "move" the NITINOL and the "washing" of the standard dies normally used for steel tools fabrication was very rapid and excessive. These preliminary forging results indicated that 60-NITINOL tools could be forged on a semi-commercial basis only from special steel dies which would have to be employed in a high energy forge hammer. These considerations would result in significantly more costly tools.

With the abandonment of close-die forging as a means of making 60-NITINOL tools the thought of cast tools was originated. Table I data tended to confirm the potential of casting 60-NITINOL tools. The "arc-cast" alloy specimens used in this property study, with their larger and less uniform grain structure, showed the same precipitation hardening and limited elongation coupled with only a modest reduction in strength. Based upon the known compatibility of molten pre-alloyed Ni-Ti alloy with graphite <sup>(1-4)</sup> it was decided to pursue a program of making and testing cast 60-NITINOL tools. The present phase, as reported in this report, is the third in the series of investigations into the casting and evaluation of NITINOL tools. The first two phases were logical and necessary steps to the present work and were previously reported<sup>(5)</sup>.

- 
1. Buehler, W., "Method for the Formation on an Alloy Composed of Metals Reactive in Their Elemental Form with a Melting Container," U.S. Patent No. 3,529,958; 22 Sept 1970.
  2. Buehler, W., "Methods of Forming and Purifying Nickel-Titanium Containing Alloys," U.S. Patent No. 3,508,914; 28 April 1970.
  3. Buehler, W., "TiNi Cast Product," U.S. Patent No. 3,672,879; 27 June 1972.
  4. Buehler, W., "Method for Casting High Titanium Content Alloy," U.S. Patent No. 3,679,394; 25 July 1972.
  5. Buehler, W. et al, "Preliminary Study Into Shell Mold Casting of Nominal 60-NITINOL Alloy," NOLTR 73-134, 12 July 1973.

## II. METALLURGICAL AND CASTING CONSIDERATIONS

Early metallurgical investigations found that pre-alloyed near-stoichiometric TiNi alloys were somewhat inert in the molten state in reasonably pure graphite (e.g., ATJ type) crucibles. The inertness was surprising particularly in light of the extreme reactivity of both molten elemental Ti and Ni with carbonaceous materials. This lack of reactivity of the molten TiNi-base alloy was strong evidence for the stability of the intermetallic bond. Reactivity with the graphite crucible was negligible until a temperature in excess of 200°C over the melting temperature was reached. This 200°C superheat without reaction was sufficient to provide ample melting flexibility for alloying followed by casting.

Initial casting efforts were performed in machined graphite molds. However, while this confirmed the suitability of graphite as a mold material it was abandoned because of mold cost and the thermal inflexibility of this molding which resulted in some shrinkage porosity.

Following the use of pure graphite molding it was logically concluded that a mold of the more conventional silica type might be employed provided its surface could be covered with a continuous coating of graphite. Two possible silica sand casting schemes were considered. They were "shell mold" casting and precision casting. Both would provide detail definition and the surface smoothness required. Economics dictated the use of shell molding.

Initially, ready-made shells were obtained from the Lynchburg Foundry, Lynchburg, Va. These were sprayed or dip-coated with a water-graphite (Aqua Dag) emulsion that formed a continuous layer of graphite when dried. These coated molds were placed in the chamber of the vacuum melting furnace and molten 60-NITINOL was poured into the cavity. This portion of the preliminary casting investigation was previously reported<sup>(5)</sup>. From this investigation

---

5. Buehler, W. et al, "Preliminary Study Into Shell Mold Casting of Nominal 60-NITINOL Alloy," NOLTR 73-134, 12 July 1973.

certain important conclusions were drawn:

1. No significant reaction between the graphite-coated standard shell mold and nominal 60-NITINOL was experienced.
2. Microscopic examination and hardness measurements of the cast alloy adjacent to the graphite-coated shell mold revealed little if any effect from the close association during solidification.
3. Mold outgassing and any localized graphite coating break-through appeared to be inconsequential. The former was not observed by the melter during pouring and the latter if it occurred must have been self-healing in the sense that the phenol-formaldehyde binder of the shell mold likely decomposed and provided a fresh carbonaceous surface.
4. Surface finish and definition was suitable for cast tool production.

With these definitive conclusions from the phase two study<sup>(5)</sup> a program was initiated to actually cast representative EOD tools from 60-NITINOL. The representative tools selected were a hammer, a plier and a wrench. They provide tool forms of varying degrees of casting difficulty and tools that would experience a spectrum of stressing and strain-rate loading in actual use.

---

5. Buehler, W. et al, "Preliminary Study Into Shell Mold Casting of Nominal 60-NITINOL Alloy," NOLTR 73-134, 12 July 1973.

### III. PATTERNS AND SHELL MOLDS

Shell molding involves a mixture of phenol-formaldehyde with silica sand. The mixture was brought into intimate contact with a heated metallic pattern causing the phenol-formaldehyde binder to soften and melt and bond adjacent silica particles in a fixed position. The shell mold was then stripped from the metallic pattern and another shell mold was made by repeating the process.

For this study suitable metallic patterns were made by machining away a symmetrical half of actual steel tools to provide half-sections. These half-sections were fastened at their machined surface to flat aluminum plates. To these tool forms were added the half-section pouring cup, gates, sprue, vent tubes, guide pins, etc. A typical half-section pattern suitable for producing a wrench mold is pictured in Figure 1.

Heating this metallic half pattern to 215°C and piling the phenol-formaldehyde-sand mixture on the hot pattern causes a somewhat uniform thickness mold half to be formed. The fusion of the binder provides the shell mold section with sufficient durability to be easily handled. A typical half wrench shell mold is shown in Figure 2. Two of these mating halves were required to provide the complete wrench mold. Prior to assembly the mold cavities were completely coated with a continuous thin layer of graphite.

Following coating the mold halves were assembled using the guide pin holes in the shell to provide alignment. Normally a thick silica-water mixture would be painted on the mold edges to prevent escapement of molten alloy during casting. This potential furnace chamber contaminant was omitted because of the subsequent vacuum casting of 60-NITINOL. This omission did result in some subsequent pour through of the molten alloy.

Finally the graphite-coated and clamped mold halves were placed in a steel container of sufficient size to accept the total shell mold. To position the shell, strengthen its walls and provide suitable heat transfer, metallic shot was poured completely around the shell, filling the steel container.

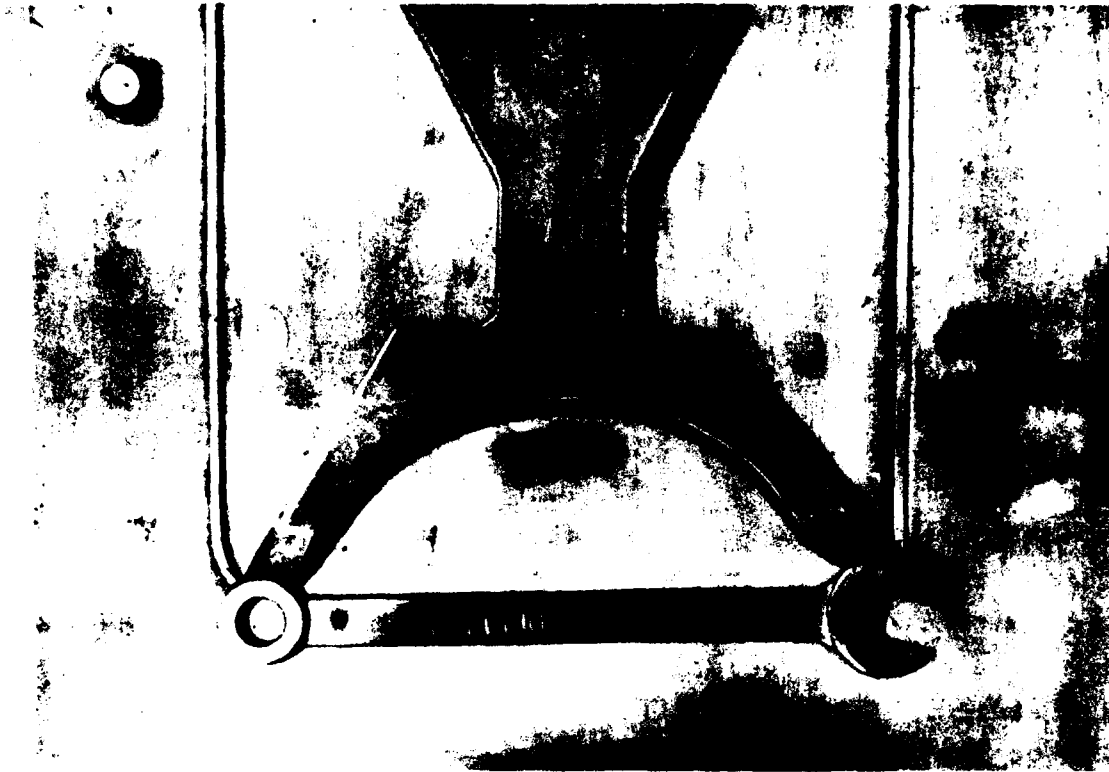


Fig. 1 One half of the wrench pattern used to produce the wrench shell mold. Note the half section of the pouring cup, sprues and vents.

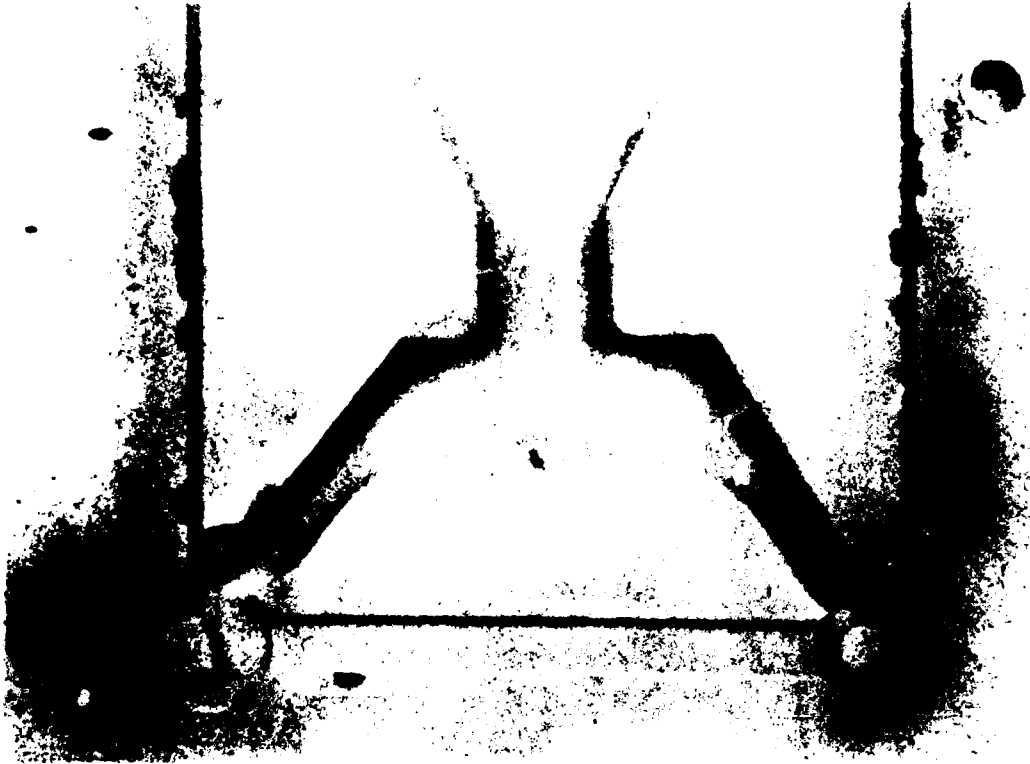


Fig. 2 A typical mold half used to cast the 60-NITINOL wrench. The cavity of this mold is completely coated with a thin graphite layer prior to assembly.

NSWC/WOL/TR 76-81

At this stage the shell mold assembly was ready to be placed in the vacuum furnace chamber, outgassed and receive the molten 60-NITINOL alloy.



#### IV. VACUUM CASTING

The TiNi-base alloys are very susceptible to rapid oxygen and nitrogen contamination if heated above about 1100°C in an air atmosphere. As a result the melting and/or alloying was conducted in a vacuum induction melting furnace. Only dry argon or helium gases were used to "backfill" when rapid outgassing of the elemental sponge titanium could produce a melt boil over.

The same unit employed for melting and/or alloying was used to perform the actual casting operation. Specifically, a prealloyed nominal 60 weight percent nickel-titanium alloy was charged directly to the ATJ graphite crucible. To this was added a small amount of elemental sponge titanium. When alloying occurred the resultant composition was approximately 58 weight percent nickel. Prior studies indicated that this composition was adequately precipitation hardenable. This composition also provides some toughness and ductility advantages over the 60 to 62 weight percent compositions.

Along with the above alloy charges the shell mold assembly was placed in the same vacuum chamber. Then with the chamber closed and evacuated, the chemically adjusted alloy was heated to the molten state. During fusion under vacuum, care was exercised to prevent any rapid outgassing of the molten alloy and possible "boil over." A suitable thermocouple sheathed in a graphite tube was used to determine the melt temperature, particularly just prior to pouring the casting.

When the alloying was completed and the molten bath temperature reached a superheat of about 200°C over the melt temperature, the melt was poured rapidly into the pouring cup of the shell mold. Early pourings resulted in some "washing" of the shell mold pouring cup area. This effect was later minimized by placing a thin graphite lining in the cup area as an integral portion of the mold assembly. With the total mold design fixed, the alloy composition and the pouring temperature established, several castings of the wrench, pliers components and the ball-peen hammer were made. These are shown in various stages of completion in Figures 3 through 8.

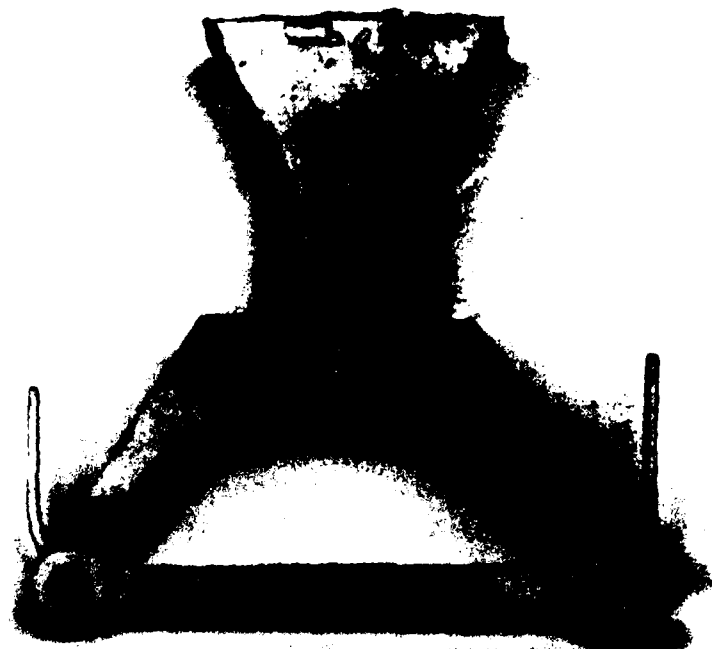


Fig. 3 Shell-mold cast assembly of the 60-NITINOL wrench. Note the limited evidence of gassing (surface pockmarkings) and the relatively high degree of liquid alloy fluidity as evidenced by the height of the molten metal in the vent holes.

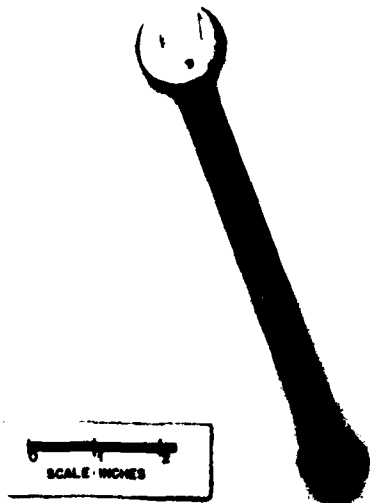
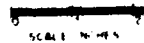


Fig. 4 Cast 60-NITINOL wrench sanded and the "socket" portion electric discharge machined to precise shape.



Fig. 5 Cast 60-NITINOL hammer head sanded and mounted on a standard glass reinforced plastic handle.



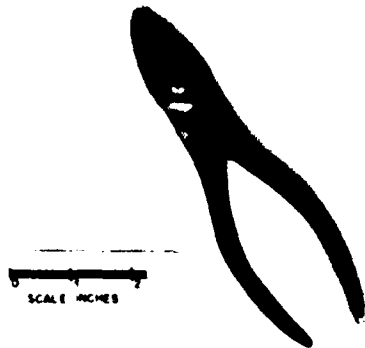
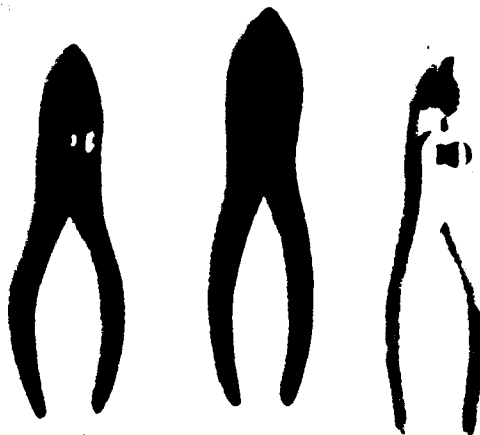


Fig. 6 Cast 60-NITINOL  
pliers sanded  
and assembled.

Fig. 7 Comparison of 60-  
NITINOL (left),  
beryllium-copper  
(center) and steel  
pliers (right)



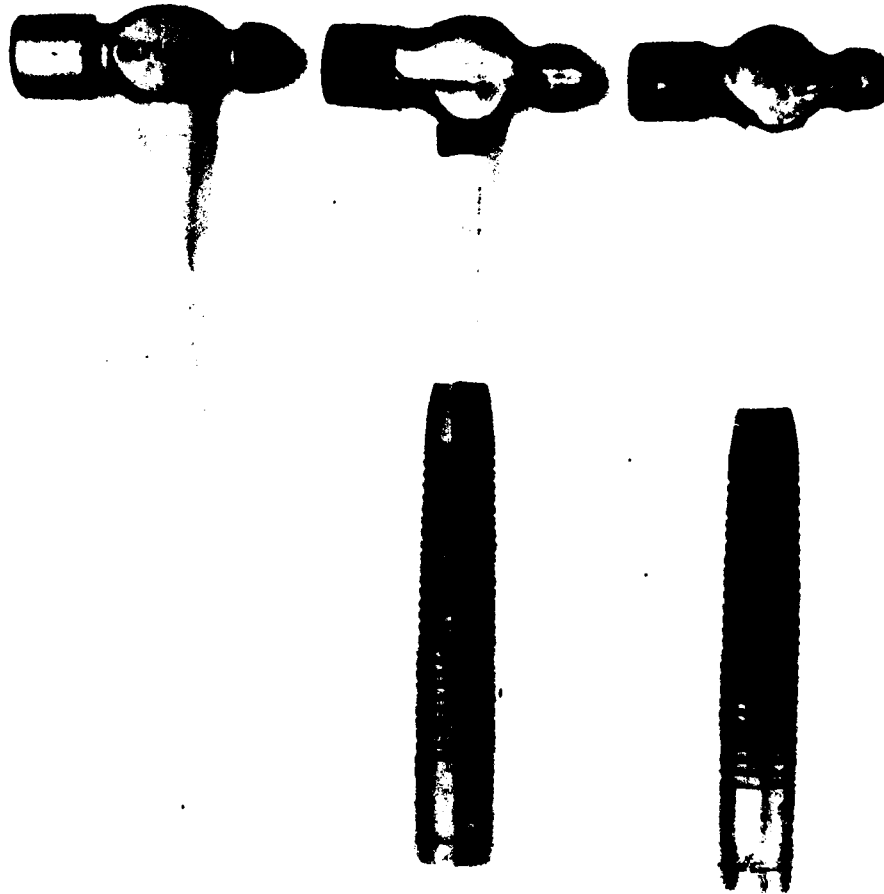


Fig. 8 Comparative photo showing the beryllium-copper (Berylco) hammer on left, steel hammer in the middle and the cast 60-NITINOL hammer on the right.

## V. ALLOY MICROSTRUCTURE

A casting study using a relatively new molding technique requires some microstructural analysis of the cast alloy material. Figures 9 through 14 provide a rather complete microstructural analysis of both the present cast tools and previous cast discs, also produced by shell mold casting.<sup>(5)</sup>

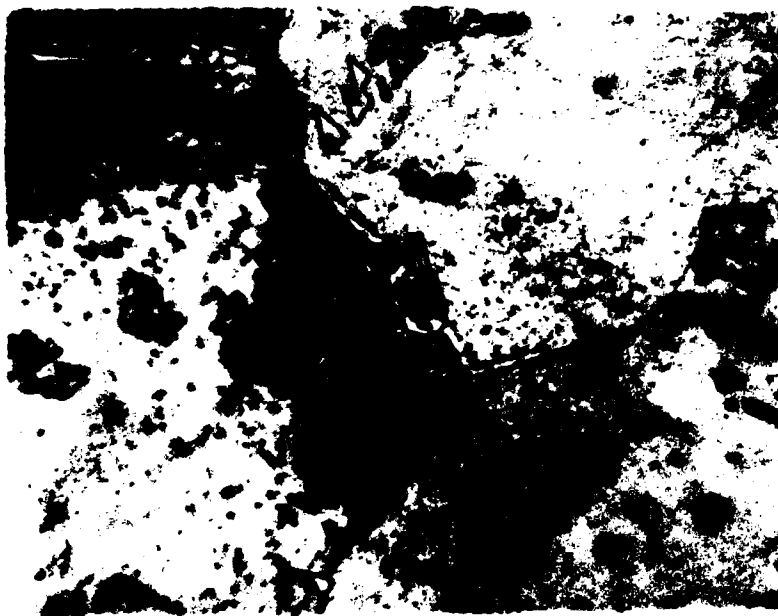
The major observable differences between the two sets of castings are two:

1. The quantity of the "mottled structure" present in the cast tools appears significantly greater than that observed in the cast discs.<sup>(5)</sup> Compare Figures 9 and 10 (tool castings) with Figure 13 (disc castings).
2. The observable background precipitate seen in the present "as cast" and heat treated (980°C) specimens as compared with the "as cast" discs similarly heat treated. This comparison can be seen by observing Figures 11 and 12 (tool castings) and matching them against Figures 13 and 14 (disc castings).

From the microscopic evidence one must conclude that the "mottled structure" and the crystallographic orientation amplified above are not artifacts of the etching procedure but real evidence of a microstructural difference. Intuitively one would have to suspect that these added microstructural features do not enhance ultimate mechanical performance. This conclusion was based upon many prior microstructural observations of the nominal 60-NITINOL material alloyed and cast under very controlled conditions. From a cursory view the "mottled structure" was probably the result of some contaminant reaction. The crystallographic precipitation was strongly reminiscent of some earlier studies by one of the authors (W. J. B.) on the diffusion of a surface oxide through sheet NITINOL when heated at 1100°C even in a helium atmosphere. The latter oxide diffusion markedly lowered the ductility of the sheet.

---

5. Buehler, W. et al, "Preliminary Study Into Shell Mold Casting of Nominal 60-NITINOL Alloy," NOLTR 73-134, 12 July 1973.



710X

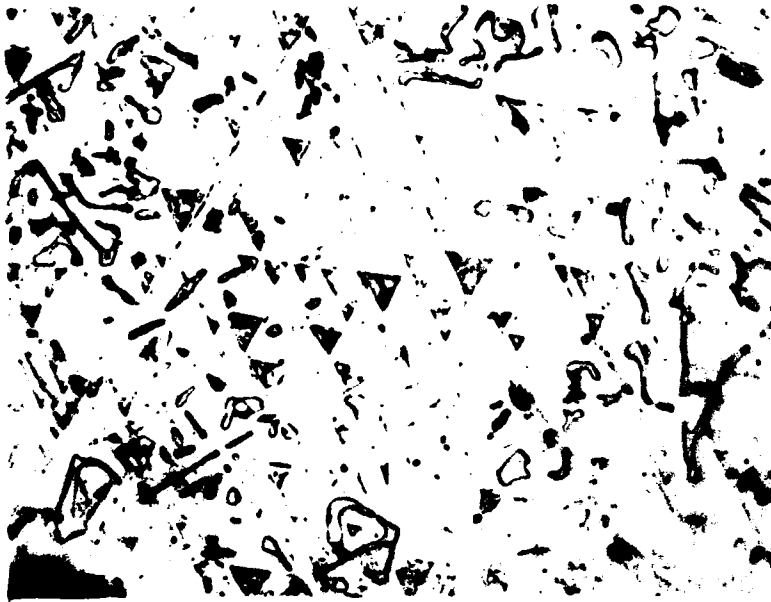
- A. A mottled two-phase zone adjacent to the grain boundary was uniformly observed. Also some isolated  $\text{TiNi}_3$  phase is observed in the "saturated"  $\text{TiNi}$  matrix phase.



710X

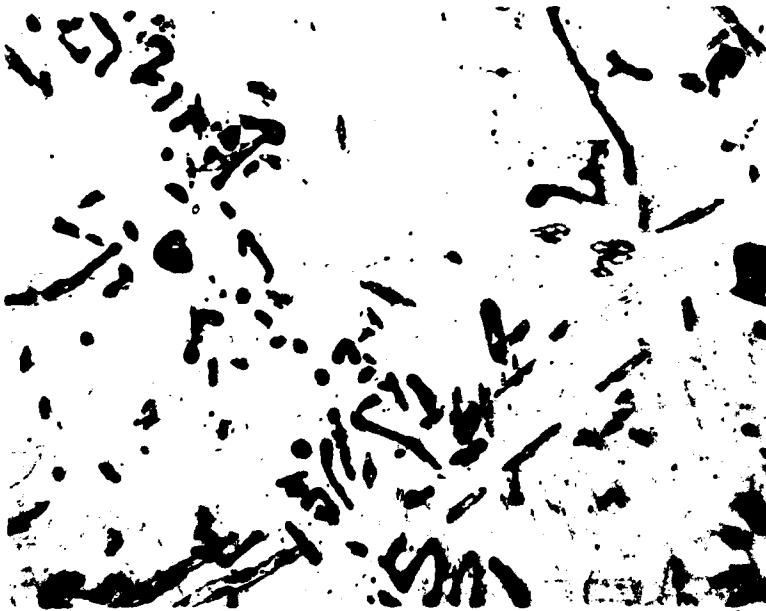
- B. This area microstructure is more "typical" of excess  $\text{TiNi}_3$  phase in a "saturated"  $\text{TiNi}$  matrix.

Fig. 9 Photomicrographs taken in different, but "typical," sections of the shell mold "as cast" 60-NITINOL material.



710X

- A. Heating time-48 hrs  
Microstructure changes give evidence of more  $\text{TiNi}_3$  phase precipitation along certain crystallographic directions and the general coalescence of the darker phase of the mottled structure shown in Figure 9.



710X

- B. Heating time-96 hrs  
Note advancement of coalescence of the  $\text{TiNi}_3$  and darker phases.

Fig. 10 Specimens taken from the 60-NITINOL pliers melt and given subsequent heat treatments (argon atmosphere) at  $980^\circ\text{C}$  for varying time periods followed by furnace cooling.





710X

Fig. 11 Continued heating for a total of 144 hours at 980°C (argon atmosphere) of the 60-NITINOL pliers melt followed by a furnace cool. Principal change is observed in the general size of the coalesced phases. Some crystallographic orientation still persists in the matrix phase. The coloration variation of the matrix phase from that seen in Figures 9 and 10 is an artifact caused by minor etching variation.



- A. Microstructure shows a "clean appearance" with the  $\text{TiNi}_3$  phase uniformly distributed in a saturated TiNi matrix.

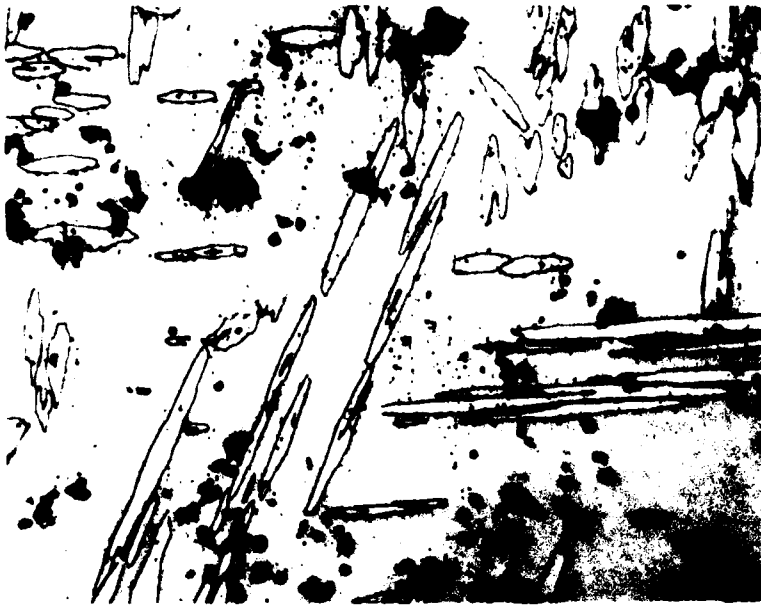
710X



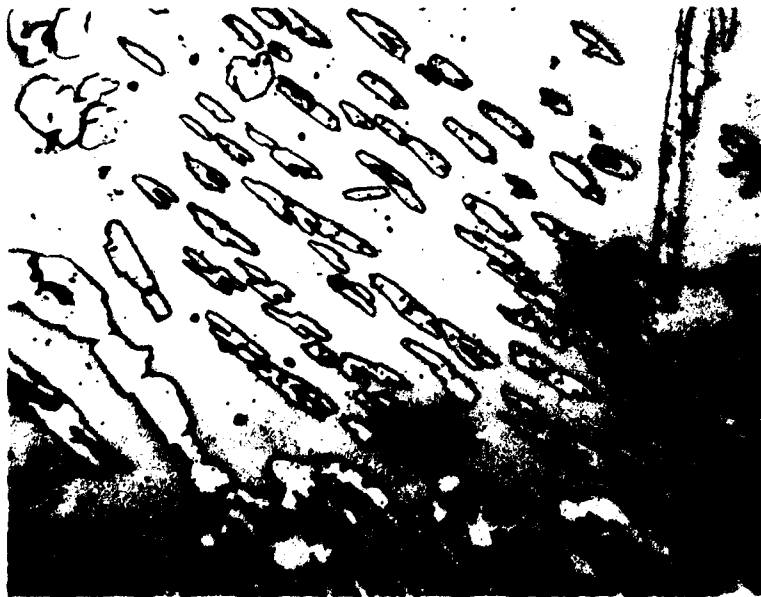
- B. Some minor occurrence of the "mottled structures" observed in Figure 9(A)

710X

Fig. 12 "As Cast" 60-NITINOL specimens taken from the cast discs.



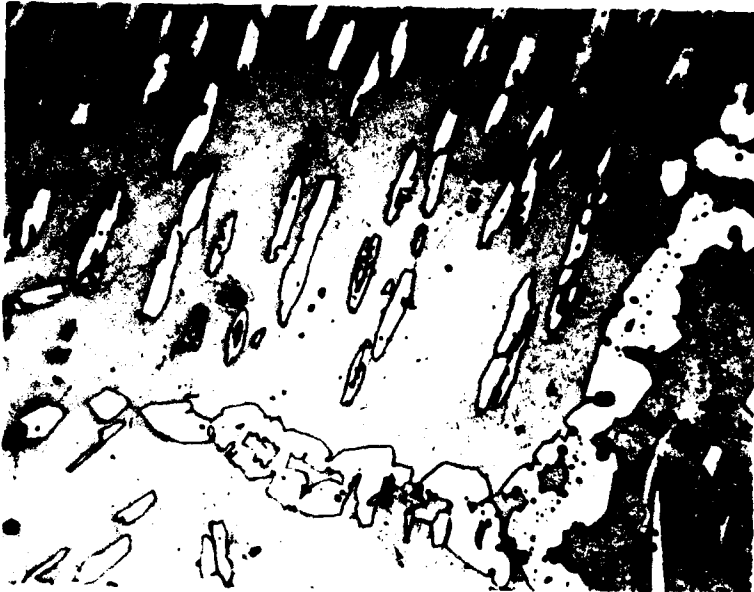
A. Surface sanded to evaluate possible surface oxide diffusion. No difference was observed.



B. "As Cast" surface.

710X

Fig. 13 Disc specimens after a 48 hour heat treatment (argon atmosphere) followed by a furnace cool. Major changes over the "as cast" structure were in the growth of the  $TiNi_3$  phase. Black particles are  $TiC$ .



A. Surface sanded to evaluate possible surface oxide diffusion. No difference was observed.

710X



B. "As Cast" surface.

710X

Fig. 14 Disc specimens after a 96 hour heat treatment (argon atmosphere) followed by a furnace cool. No observable difference appears to have occurred with the added 48 hours at 980°C, see Fig. 13 for comparison.

As a result of these observations, the present "as cast" or "as cast" and heat-treated 60-NITINOL has to be suspect. Subsequent mechanical tests of the cast tools possessing these microstructures may give significantly lower results than what might have been obtained on a purer material.

How do these possible contaminations occur? One can only conjecture but a likely source may lie with the possible washing of the pouring cavity and/or sprue areas during the pouring phase of casting. Graphite inserts were employed to minimize this effect but they may not have been totally successful.

## VI. HEAT TREATMENT

In addition to the observed microstructural changes that occurred in the 60-NITINOL with heat treatment, hardness changes were also monitored. Based upon prior experience with the precipitation hardened 60-NITINOL, the higher "as cast" hardnesses ( $>40R_C$ ) seen in Figure 15 were expected. Further, experience has shown that a relatively few hours at a temperature in excess of  $950^{\circ}\text{C}$ , followed by a furnace cool, can lower the hardness of 60-NITINOL to a "base" hardness in the high 20's to mid 30's Rockwell-C range. Since no data is provided between 0 and 48 hours the actual curve shape may be somewhat misleading. In fact shorter time data (at the  $980^{\circ}\text{C}$  temperature) may have revealed a major hardness drop to near "base" hardness within the first two hours.

One anomaly appears to be the continued drop in hardness of the alloy specimen starting at 45.5 Rockwell-C in the "as cast" condition. This specimen is considerably different in its behaviour from the generally accepted performance of a nominal 60-NITINOL material. The basis for this continued drop in hardness was unknown at the time this report was prepared.

Based upon these hardness results all cast 60-NITINOL tools were annealed for 146 hours at  $984^{\circ}\text{C}$  in an inert controlled atmosphere.

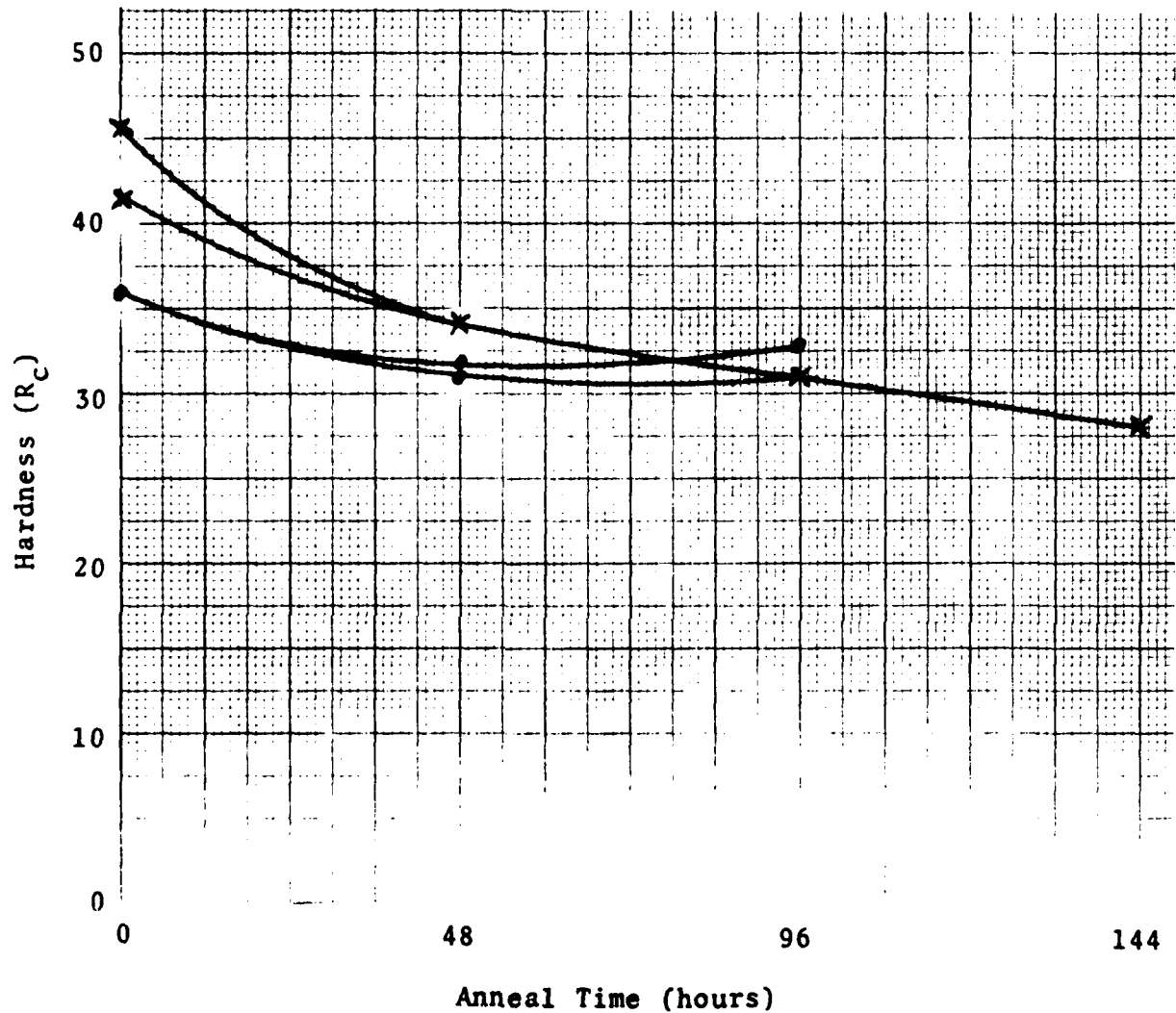


Fig. 15 The effects of heat treating various shell mold cast 60-NITINOL specimens for increased time periods at 980°C in a argon atmosphere. Cooling was at the rate of the heat treating furnace in every case.

## VII. POST-CASTING TOOL FINISHING AND ASSEMBLY

Shell molding proved to be an effective means of casting a suitably accurate shaped tool. Complexity of shape, within the tools cast, appeared to present no problems. The 60-NITINOL material had sufficient liquid fluidity to provide adequate shape definition and smooth surface finish.

However, regardless of the cast surface finish, some cleanup of certain tool surfaces was mandatory. For example, the faces of the flat and peen ends of the hammer are such surfaces. The wrench required sanding of its flat end, or working, surfaces. Finally, the plier halves required some surface finishing on the adjacent mating faces.

Additional machining was required on the "box" end of the wrench, see the case form in Figure 3. Initial efforts to cast the multi-notched "box" end were unsuccessful because of shell molding problems. On parting the mold from the pattern these delicate edges tended to crumble away. To avoid this problem and not consume an inordinate amount of time in its solution, the "box" end of the wrench was cast circular and undersize (see Figure 3). The hole was then enlarged to the proper shape by electrical discharge machining (ELOY process), see "box" end of Figure 4.

Finally, the plier halves required machine finishing on their shearing cutting edges and in the reinforcement of the gripping serrations. The former was done by hand surface grinding while the latter required cross milling using a carbide cutter.

Following all machining operations on the three annealed 60-NITINOL tools (see Table II) they were preferentially flame hardened to enhance their tool performance. These pre-assembly oxy-acetalene flame hardening heat treatments were performed in air and resulted in the changes given in Table III.

Assembly was required only of the hammer and pliers. The hammer head was attached to the glass reinforced handle by using a combination of epoxy cement and a suitable non-magnetic wedge.



TABLE II

DATA ON FULLY ANNEALED\* 60-NITINOL  
AND STANDARD HARDENED STEEL TOOLS

| TOOL   | HARDNESS (ROCKWELL) <sup>†</sup>     |  |
|--------|--------------------------------------|--|
|        | 60-NITINOL                           | STEEL                                  |
| Pliers | 66R <sub>A</sub> (32R <sub>C</sub> ) | 75.5R <sub>A</sub> (49R <sub>C</sub> ) |
| Wrench | 60R <sub>A</sub> (20R <sub>C</sub> ) | 71.5R <sub>A</sub> (42R <sub>C</sub> ) |
| Hammer | 63R <sub>A</sub> (26R <sub>C</sub> ) | 72R <sub>A</sub> (43R <sub>C</sub> )   |

\*Heated 146 hours at 980°C in a controlled argon atmosphere,  
followed by furnace cooling.

†Rockwell values actually measured on the A-scale.

TABLE III

## 60-NITINOL TOOL FLAME HARDNESS RESULTS

| TOOL   | PORTION HARDENED                        | HARDNESS (ROCKWELL) |                  |
|--------|---|---------------------|------------------|
|        |   | ANNEALED            | HARDENED         |
| Pliers | Cutting edges and<br>gripping serations | 32R <sub>C</sub>    | 41R <sub>C</sub> |
| Wrench | Box and open ends                       | 20R <sub>C</sub>    | 40R <sub>C</sub> |
| Hammer | Flat Face End-                          | 26R <sub>C</sub>    | 50R <sub>C</sub> |
|        | Ball Peen End                           |                     | 54R <sub>C</sub> |

One immediate observation on the assembled hammer was its "light headedness." This was the result of casting the 60-NITINOL head the same size as a steel head while the 60-NITINOL has a density 20% less than steel.

To facilitate assembly of the pliers, the pivot pin was machined from wrought non-magnetic titanium alloy. The actual pin design and attachment was similar to that used on steel tools. One immediate observation made on the assembled pliers was the ability under normal gripping of the handles to obtain some noticeable elastic flexure. This flexure, noticed to a minor extent in steel pliers, is an almost direct indication of the elastic modulus (E) of the tool material. Steel with an E value of  $200 \times 10^9$  newton/meter<sup>2</sup> ( $29 \times 10^6$  psi) presents a much stiffer material under load than the  $110 \times 10^9$  newton/meter<sup>2</sup> ( $16 \times 10^6$  psi) exhibited by 60-NITINOL.

These observations regarding lightness and flexure were not overlooked early by the present investigators. However, project restraints would not allow increased section sizing and design to be applied to the 60-NITINOL tools to provide equivalent flexure and weight compared with their steel tool counterparts. Some of these same design considerations have a definite effect on test and evaluation results. These design related results must be considered in making test comparisons with steel tools.

## VIII. TEST AND EVALUATION

### Non-Magnetic Evaluation

Magnetic effects tests as set forth in MIL-M-19595B were performed on all 60-NITINOL tools after each heat treatment as well as before and after each mechanical test. The forged steel and beryllium-copper tools were evaluated when received and after each mechanical test.

Both the 60-NITINOL and the beryllium-copper tools passed the magnetic effects tests at the 11.4cm (4 1/2 in.) test distance with signatures of less than one nanotesla (0.01 milligauss). The steel tools, of course, failed the magnetic effects tests.

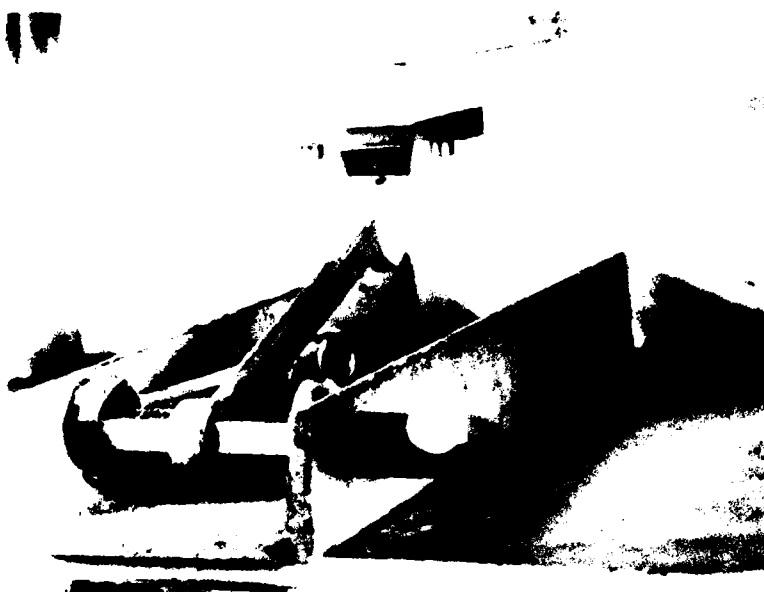
### Mechanical Evaluation

The mechanical tests were made in accordance with Federal Specifications GGG-H-86c for the hammer, GGG-P-471c for the pliers and GGG-W-636d for the combination wrench. A Tinius Olsen tensile testing machine was used to produce the measured test load for each tool test. Figure 16 shows the test fixtures which positioned the wrenches and pliers during the mechanical tests.

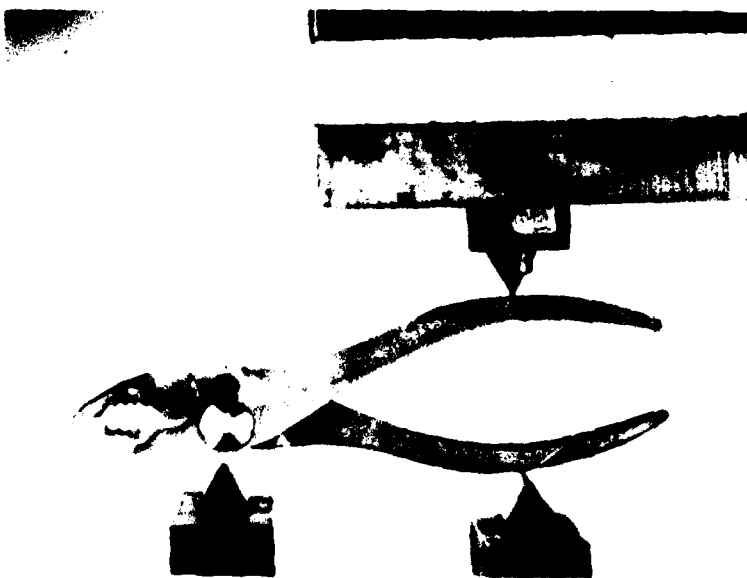
### Combination Wrench Tests

Bending moment for each end of the combination wrench was measured to the point of tool failure or until the wrench handle deflected 5 cm (2 in.), the maximum travel allowed by the test fixture. The bending moment was calculated from the product of the applied test load times the distance between the center of the fixture hexagonal mandrel and the line of action of the load. Shallow V-grooves were machined into the wrenches to produce points for the application of the test load. The load was applied to the wrench V-grooves through hardened knife edge bearing surfaces.

Figure 17 shows the combination wrenches after the mechanical tests and Table IV describes the test results. Each of the two beryllium-copper wrenches underwent only a single test. The box end of the handle fractured during one test and one side of the open

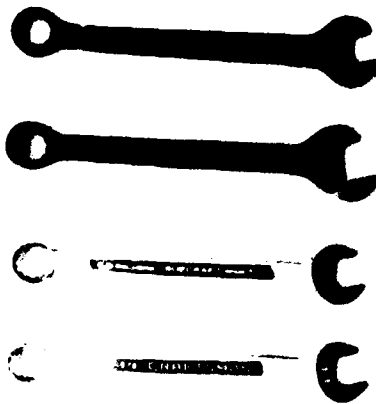


A. Wrench test method.

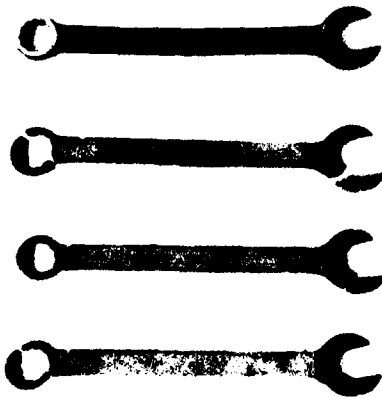


B. Pliers test method.

Fig. 16 Standard test fixtures with 60-NITINOL tools under load.



A. Beryllium-copper  
wrenches (top pair)  
Steel wrenches  
(Bottom pair)



B. 60-NITINOL wrenches.

Fig. 17 Tested wrenches - Failure modes for beryllium-copper and 60-NITINOL wrenches are about equivalent with the exception of the handle failure in the top beryllium-copper wrench.

| TOOL TESTED             | HARDNESS*<br>R <sub>C</sub> | TEST END    | TEST LOAD*<br>JOULE (IN LB) | HEAT TREATMENT  | FAILURE MODE   |
|-------------------------|-----------------------------|-------------|-----------------------------|---|--|
| NITINOL #1              | 28                          | Box<br>Open | 118 1045<br>46 405          | Fully Annealed  | Box end fractured<br>Spread around hex<br>stock              |
| NITINOL #2              | 49                          | Box<br>Open | 162 1431<br>68 598          | 900°C-15 min-<br>Vermiculite cool                             | Both end fractured   |
| NITINOL #3a             | 34                          | Box         | 116 1027                    | As above plus<br>700°C-1 hr.-<br>Furnace cool                 | Box end fractured  |
| NITINOL #3b             | 43                          | Open        | 84 740                      | As above plus<br>800°C-15 min.-<br>Verm. cool                 | Open end fractured   |
| NITINOL #4              | 42                          | Box<br>Open | 135 1196<br>72 633          | 700°C-1 hr.-<br>Furnace Cool<br>+700°C-15 min.-<br>Verm. cool | Both end fractured   |
| Steel #1                | 47                          | Box<br>Open | 292 2580<br>170 1508        | As Received   | Handle deflected<br>5cm (2 in). Spread<br>around hex stock.  |
| Steel #2                | 42                          | Box<br>Open | 253 2243<br>162 1430        | As Received   | Handle deflected<br>5 cm (2 in). Spread<br>around hex stock. |
| Beryllium-<br>Copper #1 | 40                          | Box         | 282 2498                    | As Received   | Handle broke near<br>box                                     |
| Beryllium-<br>Copper #2 | 40                          | Open        | 276 2444                    | As Received   | Open End Fractured   |

\*Required Test Values: Box End Test Load - 248 joules (2200 in lb)  
 Open End Test Load - 124 joules (1100 in lb)  
 Hardness Range - 40-55R<sub>C</sub>

TABLE IV. COMBINATION WRENCH MECHANICAL TEST RESULTS

end fractured in the other. The steel wrenches did not fracture, but under load, the open end spread around the hexagonal allen wrench mandrel and during the box end test, the handle deflected 5cm (2 in), the test fixture travel limit.

The four 60-NITINOL wrenches underwent several different series of heat treatment in an attempt to optimize the mechanical properties. The top wrench in Figure 17B was in the fully annealed condition and was the only wrench to have a permanent deformation in the handle. This wrench was also the only 60-NITINOL wrench where the open end spread around the hexagonal mandrel; the others had fractures of one side of the open end. Both the top and third wrench down had single fractures of the box end while the other two wrenches had double fractures of the box end.

#### Pliers Tests

Pliers mechanical tests were performed in a manner similar to the wrench tests. V-groove notches were machined at the points of maximum handle curvature (center of handle bow) and at a point directly below the center of the slip-joint bolt with the pliers horizontal. Hardened knife edge bearing surfaces were used to position and apply the test load. The test load for all pliers was 76 joules (675 inch-pounds) which was calculated from the product of the applied test load times the distance between the center of the slip-joint bolt and the line of action of the load.

Both steel and beryllium-copper pliers withstood the required test load of 76 joules (675 inch-pounds) (see Table V) but the 60-NITINOL pliers fractured before the load was released. Figure 18 shows the 60-NITINOL pliers failure mode which was through the thinned section of one plier half.

#### Hammer Tests

Hammer tests consisted of striking tests for the hammer faces and peens. Each face and peen was manually struck twelve blows by the ball-peen of a 114 gram (4 ounce) machinists ball-peen



TABLE V  
PLIERS MECHANICAL TEST RESULTS

| TOOL<br>TESTED | HARDNESS*<br>R <sub>C</sub> | TEST LOAD*<br>INCH-POUNDS | REMARKS†  |
|----------------|-----------------------------|---------------------------|---|
| NITINOL        | 44                          | 76 joule<br>(675 in lb)   | .71cm (.281") Deflection<br>Broke before release of<br>load |
| Steel          | 49                          | 76 joule<br>(675 in lb)   | .30cm (.118") Deflection<br>.0076cm (.003") Set             |
| BeCopper       | 43                          | 76 joule<br>(675 in lb)   | .48cm (.190") Deflection<br>.0127cm (.005") Set             |

\* Required test values; Jaw Hardness - 45-60R<sub>C</sub>

Test Load - 76 joule (675 in lb)  
Maximum Deflection under load - .81cm (0.32 in)  
Maximum Permanent Set - .076cm (0.03 in)

† All pliers passed the nail cutting requirements.

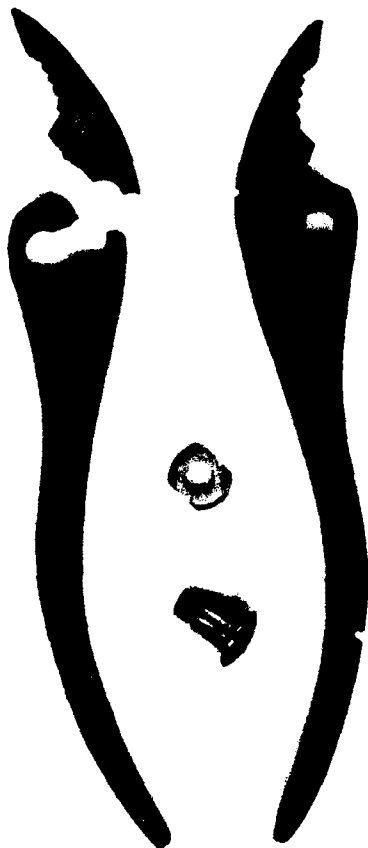


Fig. 18 60-NITINOL cast pliers failure. Note the failure mode was through the thinned section of one plier half. Low strain-to-failure coupled with the limited section thickness was responsible for the break.

hammer having a hardness of 50-60R<sub>C</sub>. Secondly, each face and peen was manually struck twelve full blows against the flat surfaced end of a rigidly supported steel bar with a hardness greater than 92R<sub>B</sub>. At the completion of these striking tests, the faces and peens were checked for chipping, cracking and spalling.

Table VI describes the results of the hammer tests. The 114 gram (4 ounce) machinist ball-peen hammer blows caused slight dimples in the 60-NITINOL test hammer, noticeable dimples in the steel test hammer and relatively deep dimples in the beryllium-copper test hammer. Values of hardness for the steel and beryllium-copper hammers below the required 50-57R<sub>C</sub> range were the cause of their excess surface dimpling. The 60-NITINOL hammer hardness was in the required hardness range and therefore suffered only slight dimpling. Light head weight of the 60-NITINOL hammer was caused by the lower density of 60-NITINOL as compared to steel and beryllium-copper.

TABLE VI  
HAMMER MECHANICAL TEST RESULTS

| TOOL TESTED | HARDNESS<br>R <sub>C</sub> | HEAD<br>WEIGHT     | REMARKS  |
|-------------|----------------------------|--------------------|--|
| NITINOL     | 54-Peen<br>50-Face         | 283gram<br>(10 oz) | Blows with 114gram (4 oz) Ball Peen<br>Caused surface dimples 0-.0076cm<br>0-3 mils deep     |
| Steel       | 43                         | 340gram<br>(12 oz) | Blows with 114gram (4 oz) Ball Peen<br>Caused surface dimples .0076-.0152cm<br>3-6 mils deep |
| BeCopper    | 41.5                       | 340gram<br>(12 oz) | Blows with 114gram (4 oz) Ball Peen<br>Caused surface dimples .038-.051cm<br>15-20 mils deep |

\* Required Test Value; Hardness 50-57R<sub>C</sub>

## IX. CONCLUSIONS

### Metallurgical Conclusions:

1. Shell-molding, using standard composition shells that are coated with a thin graphite coating produce sound 60-NITINOL tool castings with good dimensional accuracy and adequate cast surface finish.
2. Very complex cast shapes of 60-NITINOL appear possible based upon fluidity observations in this study.
3. Some reduced mechanical properties, particularly strain-to-failure, may have resulted from contamination associated with washing in the pouring cup and sprue areas during pouring.
4. Minor mold modifications to produce a "quieter" pour and minimize graphite coating washing could be simply accomplished.

### Design Conclusions:

In order to design effective 60-NITINOL tools, section thickening is necessary to provide equivalent "stiffness" to that exhibited by steel tools. This would prevent the feel of springiness found in the test pliers as well as strengthen the handles and slip-joint area. Section thickening would also strengthen the wrench box and open ends. The difference in elastic modulus (E) between 60-NITINOL and steel would solely determine the required section thickening.

Head weight for the hammer, totally dependent on density and size, can only be designed comparable in weight to the steel tool by increasing head size.

Required section thickening for the 60-NITINOL wrench and pliers would result in tools comparable in size to the larger than steel, beryllium-copper tools. Possible longer service life for the redesigned 60-NITINOL tool would have to be compared with the "off the shelf" and low cost of the beryllium-copper tool.

REFERENCES

1. Buehler, W., "Method for the Formation on an Alloy Composed of Metals Reactive in Their Elemental Form with a Melting Container," U.S. Patent No. 3,529,958; 22 Sept 1970.
2. Buehler, W., "Methods of Forming and Purifying Nickel-Titanium Containing Alloys," U.S. Patent No. 3,508,914; 28 April 1970.
3. Buehler, W., "TiNi Cast Product," U.S. Patent No. 3,672,879; 27 June 1972.
4. Buehler, W., "Method for Casting High Titanium Content Alloy," U.S. Patent No. 3,679,394; 25 July 1972.
5. Buehler, W. et al, "Preliminary Study Into Shell Mold Casting of Nominal 60-NITINOL Alloy," NOLTR 73-134, 12 July 1973.

DISTRIBUTION

|  | Copies |
|--|--------|
| Commander<br>Naval Sea Systems Command<br>Washington, D.C. 20360<br>Attn: M. Studds, Code SEA-663C-3<br>Attn: F. Henry, Code SEA-0333C | 1<br>1 |
| Commander<br>Naval Ordnance Station<br>IndianHead, Maryland 20640<br>Attn: J. R. Wilson  | 1      |
| Commander<br>Naval Explosive Ordnance Disposal Facility<br>IndianHead, Maryland 20640<br>Attn: EM J. Hamrick<br>Attn: Code XA          | 1<br>1 |
| DOD EOD Program Board<br>Code OP-03X<br>NEODF<br>IndianHead, Maryland 20640  | 1      |
| Chief of Naval Operations<br>Navy Dept.<br>Washington, D.C. 20350<br>Attn: Code NOP-03X  | 1      |
| Commander<br>Naval Sea Systems Command<br>c/o U.S. Naval Academy, Annapolis, MD 21402<br>Attn: M. Gionfriddo, Code 07013A              | 1      |
| Virginia Polytechnic Institute<br>Blacksburg, Virginia 24060<br>Attn: W. J. Buehler  | 6      |
| Lynchburg Foundry<br>Lynchburg, VA 24500<br>Attn: Larson Wiles   | 1      |
| Defense Documentation Center<br>Cameron Station<br>Alexandria, Virginia 22314  | 12     |

NSWC/WOL/TR 76-81

DISTRIBUTION (Cont.)

Copies

Naval Sea Systems Command  
Washington, D.C. 20360  
(SEA-09G32)

2

Naval Sea Systems Command  
Washington, D.C.  
(SEA03B)

1